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IGC studies of binary cationic surfactant mixtures

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Received 4 May 2007; accepted 23 July 2007

Available online 28 July 2007

Abstract

Inverse gas chromatography (IGC) has been used to measure the interaction parameter between two twin-tailed cationic surfactants. Dido-decyltrimethylammonium (DDAB) and dioctadecyltrimethylammonium (DODAB) bromides and their mixtures were used as stationary phases. IGC and DSC techniques have been used for the determination of the temperature zone of working. The activity coefficients at infinite dilution (on a mole fraction basis) were calculated for eleven probe solutes on each pure surfactant column. Values of interaction parameter between surfactants obtained at four weight fractions of the mixtures and at five temperatures are positive and suggested that the interactions is more unfavourable with the increment of DODAB concentration in the mixture. The results are interpreted on the basis of partial miscibility between DDAB and DODAB.

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Keywords: Cationic surfactant mixtures; IGC; DSC; Interaction parameters

1. Introduction

The homologous double-chained liposome-forming cationic lipids dioctadecyltrimethylammonium bromide (DODAB) and didodecyltrimethylammonium bromide (DDAB) have been some of the most investigated cationic lipids [1–5] but there is scarce literature on the phase behaviour of mixtures of these surfactants. Lipid mixtures in solution can be used for monitoring their structure and phase behaviour suitable for specific applications in science and technology. Such applications may require well-controlled properties [6].

The difference in chain length of these lipids (C_{18} and C_{12}) yields interesting characteristics to the behaviour of solutions of their mixtures which merits investigation on the interactions along the mixtures composition variation.

The formation of surfactant molecules aggregates such as spherical, rod-like or globular micelles, vesicles/liposomes, mesophases, etc., depend on the molecular characteristics of surfactants. The size of aggregates, architecture and confor-

mation of the chains depend on the Gibbs free energy of the system. The total energy results from the two main factors, the repulsive energy between polar heads and the attractive energy from the packing of hydrocarbon chains (hydrocarbon/water repulsion and van der Waals interaction between chains). These factors are not only applicable for pure surfactants but for mixtures, too.

For bicationic surfactant mixtures such as DDAB and DODAB, the polar head is the same and a non-synergic behaviour is expected. However, there are in literature several examples of steric hindrances in the hydrocarbon core that cause non-ideal behavior [7,8], even in mixtures of homologous surfactants [9].

Inverse gas chromatography (IGC) has been used in the characterisation of physicochemical properties of a number of liquid and solid materials [10–16]. This dynamic methodology has shown that measurements made under strictly controlled experimental condition would give thermodynamic information [17].

The term inverse means that the substance under study is placed as the stationary phase in a chromatographic column and the retention of a series of probe solutes is measured. The physicochemical parameters calculated from the retention data

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describe the molecular interactions between the studied material and the solutes. An advantage of this method is that it can be directly applicable for mixtures. Thus the polymer–polymer interaction parameter χ_{23} for miscible polymer mixtures was determined [18,19], although these values were different for each probe solute sample employed.

It was demonstrated that the dependence of χ_{23} on the solvent nature obtained by IGC basically is owed to the additive approximation of Flory–Huggins theory. Moreover, under certain conditions χ_{23} values could be obtained independently of the solute samples [20,21]. This method has been also applied to mixtures of polymers with compounds of small non volatile molecules, such as plasticisers and liquid crystals [22,23]. IGC has been employed for the characterisation of the thermodynamic behaviour of liquid crystals [24] through the activity coefficients, enthalpies and entropies involved in the dissolution process. Price and Shillcock [25] reported a study of the behaviour of siloxane-cyanobiphenyls with low molar mass. They also determined activity coefficients and interaction parameters of these liquid crystals [26].

DDAB is a pure double-tailed cationic surfactant that forms two lamellar phases with water that coexist at room temperature [27,28]. DODAB is also a twin-tailed cationic surfactant. The phase diagram of DODAB–water system at room temperature shows crystals of composition DODAB·2H₂O and isotropic solution [29].

In a previous study [30,31] we determined solubility parameters and HLB values of cationic surfactants.

This paper is an attempt at characterising DODAB and DDAB mixtures from a physicochemical point of view, through the interactions between the double hydrocarbon tails of both cationic surfactants obtained by IGC from retention data of the pure components and their mixtures. These studies permit determination of the interaction parameter of such surfactant mixtures.

2. Experimental

2.1. Materials

Didodecyldimethylammonium bromide (DDAB) and dioctadecyldimethylammonium bromide (DODAB) (Sigma, analytical grade) were used as received. All probe solvents (Merck) were 99% or better.

2.2. Differential scanning calorimetry

DSC was performed on a Perkin Elmer DSC 6 calorimeter, between 20 and 250 °C, with a scanning rate of 10 °C min⁻¹ and using samples of 5–10 mg for pure surfactants and 10–15 mg for materials collected over chromatography support. The instrument was calibrated with indium.

2.3. Inverse gas chromatography

Pure DDAB and DODAB and their mixtures were used as stationary phase and deposited on Chromosorb W, NAW, 60/80,

Table 1

Sorption heat (kJ mol⁻¹) and standard deviation of different probe solutes in DDAB and DODAB

	DDAB	DODAB
<i>n</i> -Hexane	28.7 ± 0.3	30.7 ± 0.3
<i>n</i> -Heptane	33.4 ± 0.4	34.8 ± 0.4
<i>n</i> -Octane	38.6 ± 0.2	39.3 ± 0.3
Benzene	33.5 ± 0.2	33.7 ± 0.4
Toluene	37.5 ± 0.4	37.6 ± 0.2
Cyclohexane	29.8 ± 0.4	31.1 ± 0.2
Methylcyclohexane	33.6 ± 0.2	33.6 ± 0.2
Dichloromethane	33.2 ± 0.3	32.3 ± 0.3
Trichloromethane	43.8 ± 0.4	43.4 ± 0.2
Carbon tetrachloride	35.5 ± 0.2	34.3 ± 0.2
Ethyl acetate	33.3 ± 0.3	32.6 ± 0.1

which was employed as solid support. The column filler was prepared using methanol as solvent in a rotary evaporator under a flow of dry nitrogen and was kept in a dry atmosphere before filling the columns (inoxidable steel pipes). The column was loaded and conditioned for 1 h at 90 °C under a flow of carrier gas. The amount of stationary phase on the support was determined by calcinations on about one g of material. The data employed in the specific retention volume computation were obtained by using a column 100 cm long, 1/4 inch external diameter, and the packing characteristics are included in supplementary material (Table 1).

The retention time measurement for each solute was performed with a Hewlett Packard, HP 6890 series, GC System, equipped with a flame ionization detector (FID). Column temperature was measured in a range between 363.15 and 403.15 K with an Iron-Constantan thermocouple placed in the direct environment of the column. The temperature stability during experiments was of ±0.2 K. The employed solutes were *n*-hexane, *n*-heptane, *n*-octane, cyclohexane, methylcyclohexane, benzene, toluene, ethyl acetate, dichloromethane, trichloromethane and carbon tetrachloride.

Nitrogen was used as carrier gas. Flow rates were measured at the beginning of each experiment with an air-jacketed soap film flowmeter placed at the outlet of the detector. Inlet pressures were measured with a micrometry syringe (through the injector septum) which was connected to an open branch mercury manometer. To ensure that the results were independent of sample size and flow rate and those measurements were being made at infinite dilution the usual checks were made [32].

Solutes were injected with 10 µl Hamilton syringes, as steam in equilibrium with pure liquid. For all the solutes and for all the range of stationary phase concentrations the peaks were symmetric. The injector was kept at 150 °C and the detector at 180 °C.

Retention times (t_R) were measured with a Chem Station system and the retention specific volumes (V_g^0) were calculated with the following relationship [32]:

$$V_g^0 = j(F_f/w)(273.15/T_f)(t_R - t_0)(p_0 - p_w)/p_0, \quad (1)$$

where j is the James–Martin compressibility correction factor, p_0 represents the outlet column pressure, F_f is the flow rate measured at pressure p_0 and temperature T_f , w is the mass of

the stationary phase into the column and p_w is the water vapour pressure at T_f ; t_0 is the dead time, which was measured by using the methane peak obtained with the FID.

3. Results and discussion

3.1. Pure surfactants as stationary phase

Chow and Martire [24] compared IGC and DSC studies on two azoxy liquid crystals and reported no measurable adsorption effects from the interface above a film thickness of 100 nm. Witkiewicz [33] reported surface orientation effects up to depth of 2 nm, but in a later work reported constant specific retention volumes above a stationary phase loading of above 5% [34]. Zhou et al. [35] in the GC and IR study of liquid crystal deposited on different types of silica have shown that for a percentage under of 7% of the stationary phase loading, the $\ln V_g$ vs $1/T$ plot did not show discontinuity. The loading used in this work was near of 10% on Chromosorb W in all the cases.

The retention diagram of $\ln V_g$ vs $1/T$ for solute probes in DODAB and DDAB coated on Chromosorb W, NAW, 60/80, is shown in Fig. 1. For both surfactants, specific retention volumes were obtained between 30 and 150 °C. On heating the solid, the retention decreases until the solid-to-liquid crystal transition is reached. Then there is a large increase in retention, which once the system phase change finished, decreases with increasing temperature. For DDAB and DODAB changes in retention are observed at 70 and 85 °C, respectively.

Specific retention volumes were fitted to the equation [32]

$$\ln V_g^0 = \Delta H_s^0 / RT + \text{constant}, \quad (2)$$

where ΔH_s^0 is the sorption heat. The values obtained for ΔH_s^0 , as well as their respective standard deviations, were calculated using Marquardt–Levenberg's algorithm [36] and can be seen in Table 1. Values of standard deviations in ΔH_s smaller than 1.5% are obtained in the regression of specific retention values vs $1/T$, although most of the values were near 1.0%.

The meaning of ΔH_s^0 depends on the physical state of the stationary phase. For a solid, ΔH_s^0 correspond to the molar adsorption enthalpy. For the liquid mesophase, it was assumed that the solute is dissolved in the stationary phase so ΔH_s^0 suit the molar solution enthalpy. DSC analysis of the transition temperature for pure DDAB and DODAB was detailed in supplementary material. ΔH_s^0 values for alkanes in DODAB are slightly greater than in DDAB values. There are smaller differences for ΔH_s^0 in cycloalkanes and very similar values between aromatics and polar. There are equal ΔH_s^0 values for trichloromethane in both stationary phases.

Chow and Martire [24] analyzed the process of transference of a solute from a gas phase to a liquid crystalline phase at infinite dilution conditions in terms of basic statistic mechanics. Three effects could influence the magnitude of the activity coefficient at infinite dilution: the effect of the potential energy of interaction, the rotational effect and the vibrational effect. The greater contribution of the potential energy of interaction term, the smaller values of the activity coefficient. Therefore, the solubility is favoured by greater solute solvent interactions.

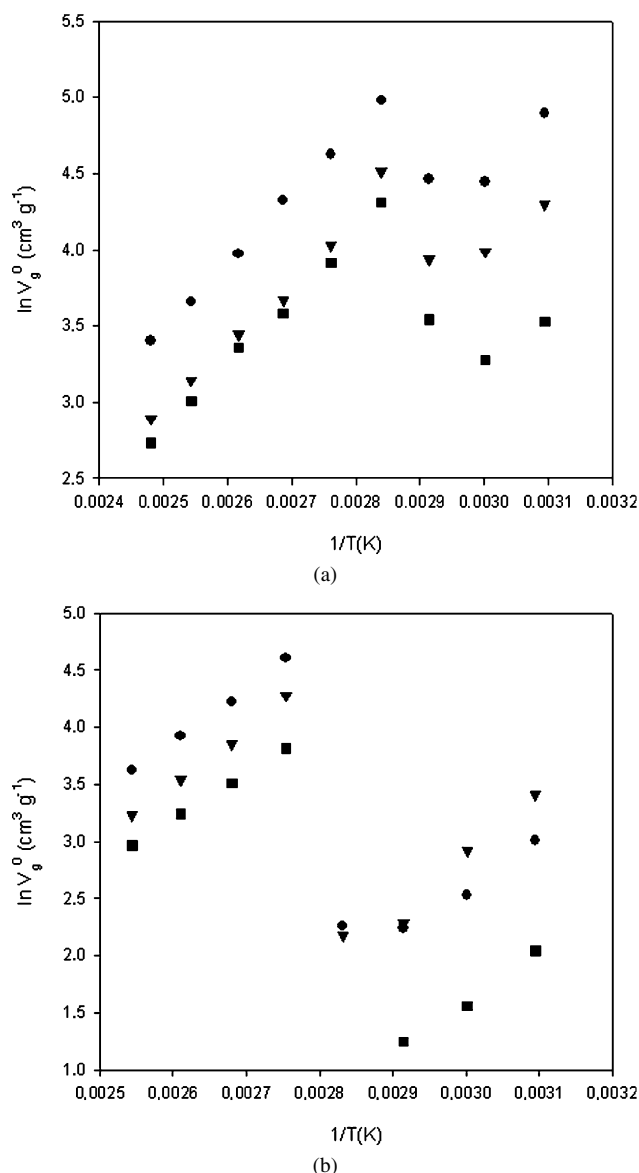


Fig. 1. Retention diagrams: (a) in DDAB; (b) in DODAB. Solutes: (●) toluene, (■) carbon tetrachloride, (▼) *n*-octane.

Rotational and vibrational effects in liquid crystal solutions are related to the restriction or loss of these movements. This would imply greater values of activity coefficients and therefore does not favour the solubility of the solute.

Values of activity coefficients at infinite dilution in terms of mole fraction were obtained by the following expression [26]:

$$\ln \gamma_i^\infty = \ln \frac{273.15R}{V_g^0 p_1^0 M_2} - \frac{p_1^0 (B_{11} - V_1^0)}{RT}, \quad (3)$$

where M_2 stands for molar mass of surfactant, p_1^0 , V_1^0 stands for vapour pressure and molar volume of pure solute. B_{11} is the second virial coefficient for solute–solute interactions. The vapour pressures were computed using Antoine equations and the coefficients were taken from Riddick et al. [37]. The solute densities at different temperatures were estimated from Dreisbach's compilation [38]. The second virial coefficient of the

Table 2
Activity coefficients at infinite dilution in terms of mole fraction (γ_1^∞) values in DDAB and DODAB at 100 °C

Solutes	DDAB	DODAB
Hexane	1.89	1.25
Heptane	2.11	1.36
Octane	2.38	1.49
Benzene	0.65	0.51
Toluene	0.82	0.64
Cyclohexane	1.27	0.86
Methylcyclohexane	1.45	0.96
Dichloromethane	0.36	0.32
Trichloromethane	0.11	0.15
Carbon tetrachloride	0.60	0.49
Ethyl acetate	1.51	1.32

solutes was calculated by Tsonopoulos's correlation using critical constants tabulated in Reid et al. [39].

The values of activity coefficients at infinite dilution (on a mole fraction basis) for some of the probe solutes in DDAB and DODAB at 100 °C are included in Table 2. Such values are slightly greater in DDAB than in DODAB. Following Chow and Martire [24], this may be either the result of a greater interaction in DODAB, or that the solute undergoes a smaller loss of rotational and vibrational motion in DODAB, or both effects simultaneously.

Increasing the length of the hydrocarbon chain in the alkane test solute with the same surfactant, the value of the activity coefficient increases due to a greater restriction to the probe solutes movement in the infinitely diluted solution. Alternatively, following Chow and Martire analysis, the effects of rotational and vibrational motion predominate over the effect of potential energy which diminishes the value of the activity coefficient for those solutes.

In solution of alkanes in other alkanes having longer hydrocarbon chain, the activity coefficient of the solute decreases when passing from solution in dodecane to that in octadecane [40] in a similar way that happens in the systems here studied. However, the activity coefficients are lower in hydrocarbons than in the more ordered liquid crystalline stationary phases we employed.

The values of the activity coefficients found for the probe solutes dichloromethane and trichloromethane in both mesophases were equal within experimental error, which would indicate that they locate in the polar head layer, which is the same in both surfactants. The values of the activity coefficients for all the test solutes in both mesophases increase slightly when temperature increases. In solvents which could be called normal the effect of the solute–solvent interactions diminishes with the increase in temperature, which is inherent to the molecular thermal motion increase and the increase in molecular separation. On the other hand, in the ordered mesophases a greater rotational-vibrational restriction would occur when temperature rises.

3.2. Surfactant mixtures as stationary phases

The Flory–Huggins interaction parameter between surfactant and test solute, χ_{12}^∞ , can be related to the specific reten-

tion volume of the probe solute, V_g^0 , by the following equation [18,32,41]:

$$\chi_{12}^\infty = \ln\left(\frac{273.15Rv_2}{V_g^0 p_1^0 V_1^0}\right) - \left(\frac{p_1^0(B_{11} - V_1^0)}{RT}\right) - \left(1 - \frac{V_1^0}{V_2^0}\right), \quad (4)$$

where v_2 stands for the specific volume of the surfactant experimentally measured in the laboratory. The values of pure components have been taken of the literature [37–39].

When a surfactant mixture is used in an IGC study the corresponding specific retention volume and specific volume of the mixtures can be used in Eq. (4), the interaction parameter obtained is called $\chi_{1(23)}$ [18]:

$$\chi_{1(23)}^\infty = \ln\left(\frac{273.15R(w_2v_2 + w_3v_3)}{V_g^0 p_1^0 V_1^0}\right) - \left(\frac{p_1^0(B_{11} - V_1^0)}{RT}\right) - \left(\phi_2\left(1 - \frac{V_1^0}{V_2^0}\right) + \phi_3\left(1 - \frac{V_1^0}{V_3^0}\right)\right), \quad (5)$$

where w_i and ϕ_i stand for the weight and volume fractions for i component in the stationary phase. V_i^0 is the molar volume of component i in the mixture.

Applying the Flory–Huggins equation of polymer solutions to a ternary system with two surfactants and one probe solute, the interaction parameter $\chi_{1(23)}$ can be related to the difference between pair interaction of probe–surfactants, χ_{12}^∞ and χ_{13}^∞ , and surfactant–surfactant χ_{23} [23]:

$$\chi_{1(23)}^\infty = \phi_2\chi_{12}^\infty + \phi_3\chi_{13}^\infty - \phi_2\phi_3\chi_{23}\left(\frac{V_1^0}{V_2^0}\right). \quad (6)$$

The dependence of χ_{23} on the solute probe nature has been considered from a different point of view [20,21,42,43]. We used Farooque and Deshpande [43] methodology who rearranged the above expression as follows:

$$\frac{(\chi_{1(23)}^\infty - \chi_{13}^\infty)}{V_1^0} = \left(\frac{\phi_2(\chi_{12}^\infty - \chi_{13}^\infty)}{V_1^0}\right) - \phi_2\phi_3\left(\frac{\chi_{23}}{V_2^0}\right). \quad (7)$$

A plot of the left side of this expression versus the first term of the right-hand side yields a lineal function from whose slope ϕ_2 can be calculated and from the intercept χ_{23} can be obtained.

The physical meaning of the slope was interpreted in terms of an effective average column composition that the solutes are probing. Fig. 2 is a plot of ϕ_2 effective calculated with Eq. (7) versus ϕ_2 values obtained from specific volume. The correlation coefficients were greater than 0.99, suggesting that the solute probe does not find a preferential site of dissolution

The values of the surfactant–surfactant interaction parameters can be analyzed as χ_{23}/V_2^0 or as χ_{23} when multiplied by V_2^0 , or as the equivalent quantity $B = RT(\chi_{23}/V_2^0)$ (in J cm⁻³) called energy density. In this work we used χ_{23} . The χ_{23} values obtained for the four studied weight fractions at the five temperatures are positives. The variation of χ_{23} with the composition in terms of fraction weights w_2 , is shown in Fig. 3. From a theoretical point of view, this parameter would be constant with the concentration. Nevertheless, frequently it has been found that

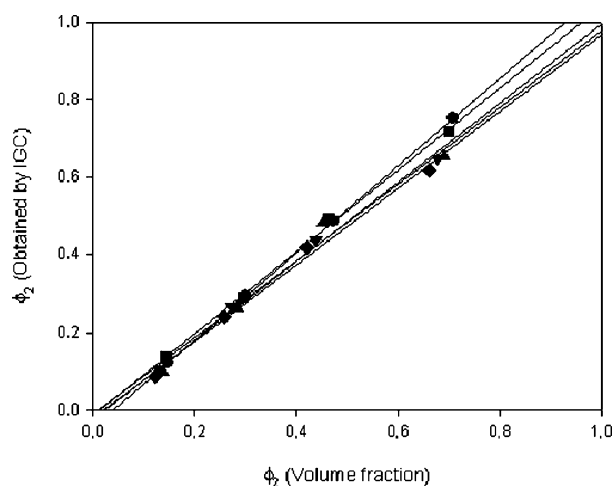


Fig. 2. Effective volume fraction (ϕ_2) of DDAB calculated from Faroque–Deshpande method. Temperature: (●) 363.15, (■) 373.15, (▲) 383.15, (▼) 393.15, and (◆) 403.15 K.

its value changes with concentration [42]. Fig. 3 shows a minimum in χ_{23} near 0.5 of weight fraction for each studied temperature. χ_{23} values are positive and suggest that the interaction between surfactants is more unfavourable as DODAB concentration (smaller values of w_2) is increased. This is in agreement with recent results from Feitosa et al. [6], who found that DDAB molecules have affinity for DODAB liposomes larger than that of DODAB molecules for DDAB liposomes. Since our experiments were done at temperatures in which lamellar liquid crystal exist, which is essentially the same mesophase that forms liposomes, this coincidence is not surprising. Since the polar head group is the same, this effect must be caused by the interaction of tails inside the hydrocarbon double layer, which is presumably scarcely influenced by the presence (liposomes) or absence (anhydrous lamellar mesophase) of water.

The incompatibility between surfactants augments as the temperature is increased and the increments in χ_{23} values are greater at greater temperatures. Positive values of χ_{23} have been obtained for mixtures of hydrocarbons *n*-tetracosane with low density polydimethylsiloxane by Patterson et al. [18] at 60 °C.

Beaucage and Stein [44] analyzed via small-angle neutron scattering analysis (SANS) the effect of the tacticity on the miscibility of mixtures of PVME (poly(vinyl methyl ether)) atactic and isotactic with deuterated polystyrene (PS) polymers. They obtained by fitting the g_{sc} (parameter interaction obtained via scattering analysis) which is temperature and composition dependent. g_{sc} values were fitted to Flory–Huggins–Staverman (FHS) equation [45], which takes into account g_{sc} composition and temperature dependency in terms of relative surface areas of the components in the mixtures. Changes in enthalpy and entropy terms that contribute to g_{sc} in the systems studied are explained by a higher order in isotactic mixtures materials with PS. Besides it may be pointed out that the more positive g_{sc} term is, the more reduced is the system miscibility.

We used the Flory–Huggins approximation, extended to ternary systems, in a system that consists of a mesophase arrangement where the hydrocarbon chain could be ordered towards the gas phase as a consequence of the arrangement of

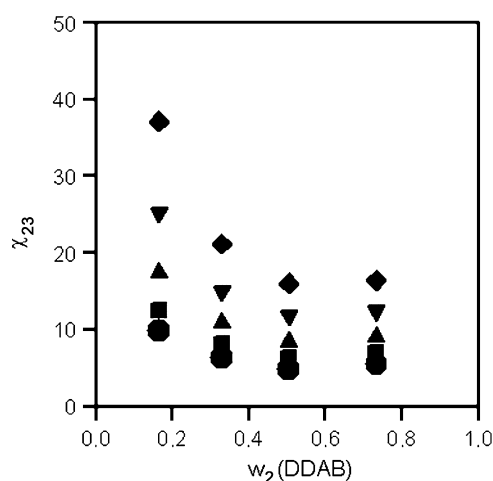


Fig. 3. Interaction parameters between DDAB and DODAB in function of weight fraction of DDAB. Temperature: (●) 363.15, (■) 373.15, (▲) 383.15, (▼) 393.15, and (◆) 403.15 K.

polar heads towards the solid support. We could think of our systems in terms of relative surface areas such as the ones used in FHS equation [45] for DODAB and DDAB mixtures. Comparing temperature and concentration effect on g_{sc} parameter interaction with χ_{23} parameter interaction obtained by IGC, a smaller order could be expected when increasing DDAB concentration (see graph in Fig. 3). By increasing DDAB concentration in the mixture there is a change in the surface area per interactive unit which could carry through to a decrease in χ_{23} parameter.

For soaps, Lecuyer and Dervichian [46] found that in a mixture where one component has n_C atoms of carbon in its tail, and the other has $n_C + x$ atoms of carbon, the smaller x is, the greater is the tendency to crystallize as if the mixture were formed by identical molecules, giving a fully miscible solid solution. This occurs for values of $x/n_C \leq 0.14$. As x/n_C is increased (between 0.14 and 0.50) the systems are able to yield stoichiometric compounds but not solid solutions for all proportions. If $x/n_C > 0.50$, soaps crystallize separately, yielding neither solid solutions nor molecular compounds. Probably solid solutions are substitutional [47]. It is possible that the same steric hindrances play a similar role in the strongly ordered lamellar mesophases. Note that in our mixtures, $x/n_C = 0.5$. If this were the case, probably two stoichiometric mixtures of DODAB and DDAB form in equilibrium as conjugate phases.

An X-ray diffractogram of usual mixtures of commercial soaps show that they crystallize as solid solutions, without a division into fractions of the constituents. This solid solution is favoured by the humidity. The mean molar mass can be inferred from the long spacing, and in general coincides with that obtained from the saponification index [47]. Recently, in a study of mixtures of cationic liposomes in water by DSC, Nile Red fluorescence and turbidity, Feitosa et al. [6] have found the formation of two populations of liposomes with different properties. The results by DSC for dilute solutions of DDAB and DODAB mixtures in water suggest that DDAB and DODAB do not form ideal mixtures. Moreover, it is shown that DODAB do not have affinity for DDAB, in concordance with our re-

sults, and their results by fluorescence show that in systems where DDAB is predominant are in liquid crystalline phase, whilst when the DODAB proportion is high the system is predominantly in gel phase. Fietosa et al. consider that two types of liposomes would exist in equilibrium, one of them rich in DDAB and the other rich in DODAB, which suggest that we are in an intermediate case.

4. Conclusions

The two twin-tailed cationic surfactants DODAB and DDAB and their mixtures were employed as stationary phases in IGC. As far as we know, there are not any other studies on solid mixtures of DDAB and DODAB without water. The thermal stability of the above mentioned phases at the working temperatures was verified by IGC and DSC. Reproducible retention values were obtained for the different probe solutes.

The obtained activity coefficients for *n*-alkane solutes were similar to those obtained for the same solutes in *n*-alkane stationary phases, but reflecting the more ordered packing of the hydrocarbon tails in the surfactant mesophases. Polar solutes show the same behaviour in all the tested stationary phases.

The surfactant–surfactant interaction parameter was dependent on the surfactant mixture composition showing that the interaction becomes less favourable when the DODAB content in the mixtures increases. This phenomenon is interpreted as a partial miscibility of both surfactants giving rise to the formation of conjugate phases. Literature data suggest that this partial miscibility also persists in mixed lamellar mesophases in the presence of water, and that it is caused by steric constraints on the packing of molecules with different chain lengths when included in a bilayer because of the constraint that the polar head groups must remain at the surface of the bilayer, as reported in literature in the case of solid soap mixtures.

Acknowledgments

This work was sponsored by CIUNT (Consejo de Investigaciones de la Universidad Nacional de Tucumán) and partially by PICT 10-14560 of ANPCyT (Agencia Nacional de Promoción Científica y Tecnológica). E.L.A. is a member of CONICET (Consejo Nacional de Investigaciones Científicas y Técnicas de la Republica Argentina).

Supplementary material

The online version of this article contains additional supplementary material.

Please visit DOI: [10.1016/j.jcis.2007.07.055](https://doi.org/10.1016/j.jcis.2007.07.055).

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